TopSpin 1.3

Users

Guide

Preface

If you are using either the DPX-250, on the sixth floor, or the DPX-400 located in B-18, you will be using TopSpin 1.3. You may well ask why the NMR facility doesn't use the same version of TopSpin on all of our Bruker spectrometers. Indeed, many people, myself included, wish that was possible. The reason that we need run two different versions of TopSpin is that the TopSpin 2.x does not support the older DPX consoles. Most NMR users will not notice many of the differences between different versions of TopSpin.

Commands that you would type at the command line appear as ***Bold, Italicized***.

(1) Starting TopSpin

Method (1):

Click on: the TopSpin Icon on your desktop

Method (2):

Open a Linux Shell/Terminal Window

Type: ***topspin***

(2) The Topspin Browser

The browser appears at the left of the TOPSPIN window and from the browser you can browse datasets, select a dataset, or open a dataset.

If the file browser is not already open, click on the Browser tab highlighted in blue (below).

The browser shows data directory tree and allows you to expand/collapse branches by clicking on the arrow to the left of each entry. Notice that the primary entries are simply the names of each NMR user.

By clicking on the icon immediately to the left of a name you will expand that directory thus revealing the datasets within that directory. In this example, I have clicked on the directory "dale" to reveal the names of several experiments I have stored in my particular data directory. By clicking on the icon immediately to the left of the experiment "15N-setup" I see each of the experiment numbers (EXPNO) within that dataset directory.
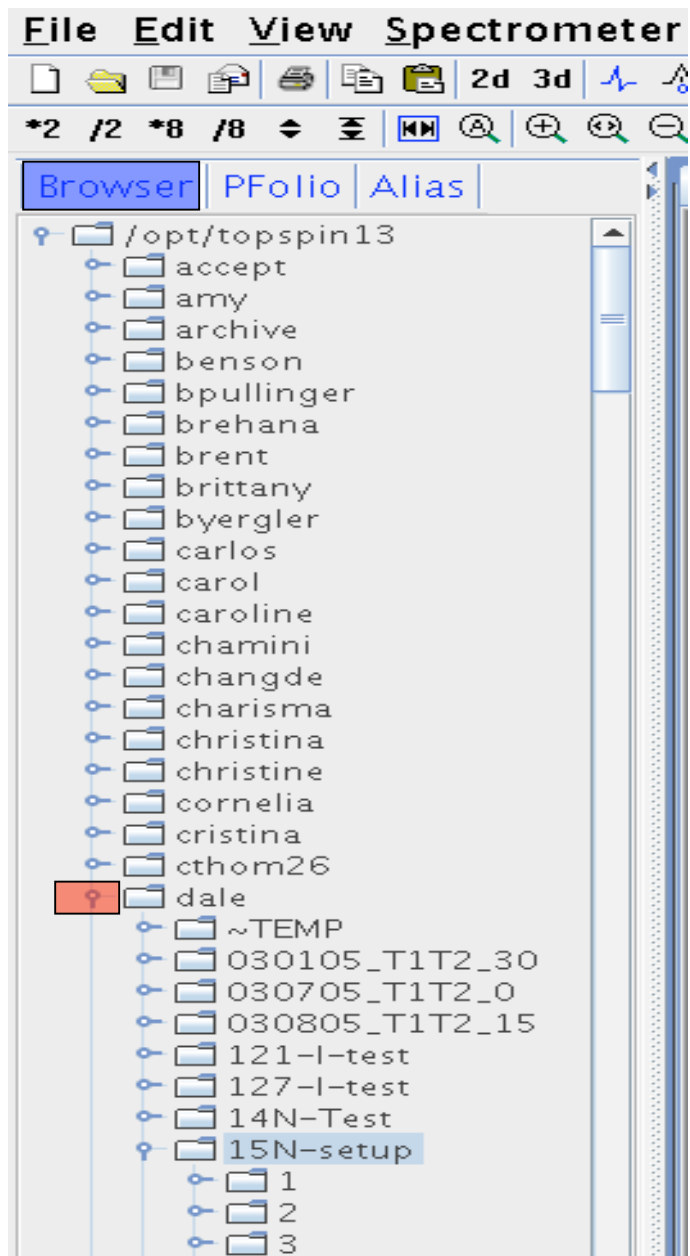
Alias: Click on the Alias tab to display the list of user defined alias names for datasets. Right-click on an entry to define, remove or interpret alias names. Personally, I don't use this feature.

(3) Opening a Dataset from the Browser

There are several ways to open a dataset. Choose the method that you like from the list below:

- Left-click-hold a dataset *name*, *expno* or *procno* and drag it into the data area. The data will be displayed in a new data window.

- Left-click-hold a dataset *name*, *expno* or *procno* and drag it into an open data window. The data will replace the currently displayed data.

- Left-click-hold a dataset *name*, *expno* or *procno* and drag it into an empty data window created with *Alt+w n*.

- Left-click-hold a dataset *name*, *expno* or *procno* and drag it into a multiple display data window.   The data will be superimposed on the currently displayed data.

- Right-click a dataset *name*, *expno* or *procno* and choose Display from the popup menu; the data will be displayed in the current data window.

- Right-click a dataset *name*, *expno* or *procno* and choose Display in new window from the popup menu; the data will be displayed in a new data window.

- Hold the *Ctrl* key and left-click several datasets to select them or hold the *Shift* key and left-click two datasets to select these two and all in between. Right-click one of the selected datasets and choose Display from the popup menu. A new window will open showing the selected datasets in multiple display mode.

> **Note:** If the current window was already in multiple display mode, the selected spectra will be **superimposed** on the currently displayed spectra.

(4) **Creating a new data set:**

There several ways that one can use to create a new dataset. Perhaps the easiest way is to

type: **Cntl N** .

A window as shown below will appear on the screen. Choose the name of your new experiment as well as the Experiment Number (EXPNO) the Process Number (PROCNO) and the USER. I recommend leaving the USER set to your normal **login ID.**   Note also that you can specify the solvent you are using.
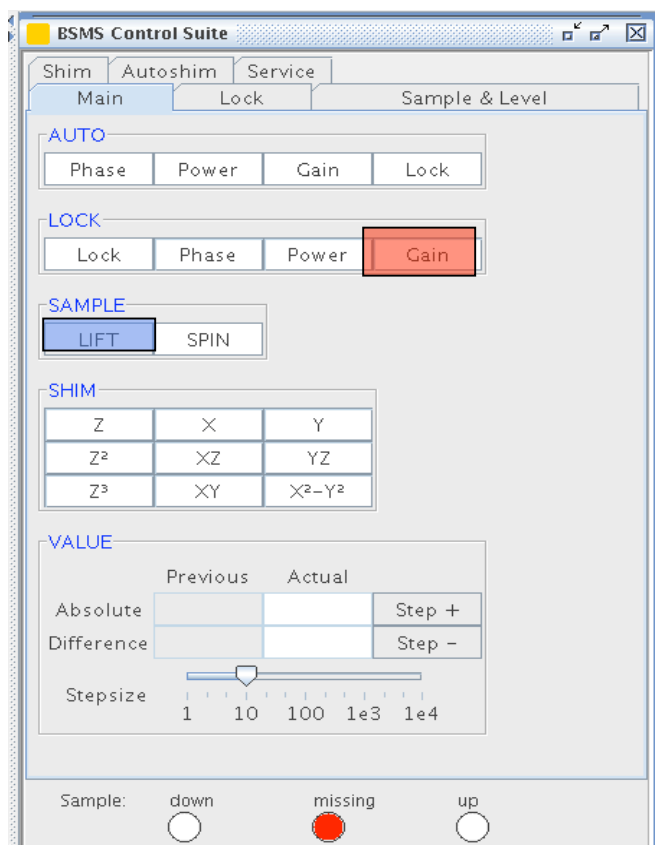
Click on: **OK** when you are finished.
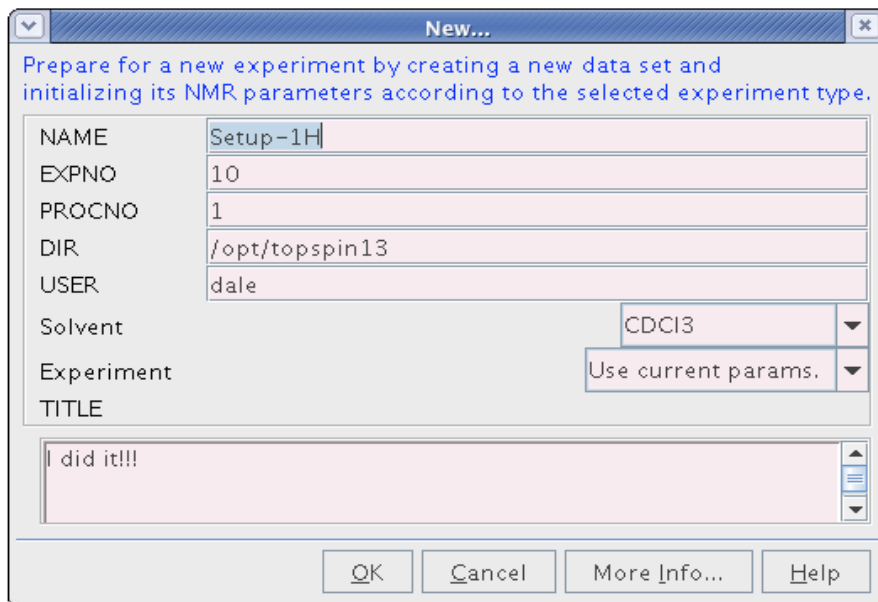
(5) Locking and Shimming the magnet on Your Sample:

Click on lock display icon highlighted in blue
(or type: *lockdisp*)
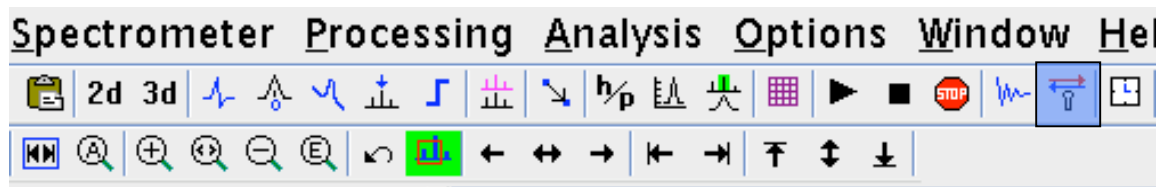
To open the bsmsdisp suite window as shown below.

Type: *bsmsdisp*

- Left-click on Lift highlighted in blue above.

- Insert the sample tube into a spinner and adjust the sample depth.

- Place the sample atop the magnet after ensuring that you can hear the lift
- air.

- Left-click on Lift a second time to lower the sample into the magnet.

- Type: **Lock**
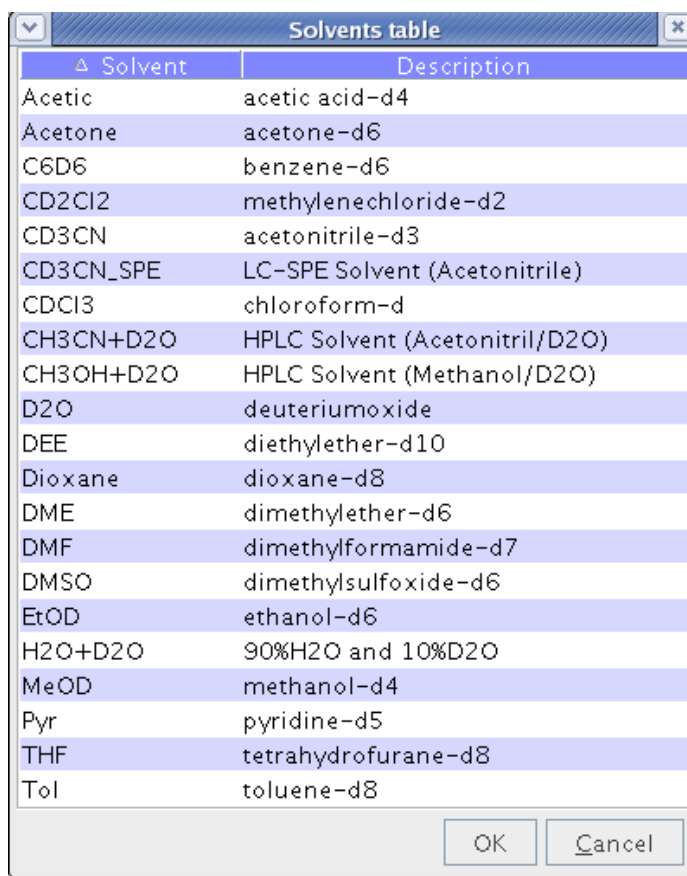
- Select your solvent from the popup window shown below.

Type: **rshm shimlast**

Optimize the X,Y,Z, and Z^2 as follows:

- For example, Left-click on the Z-shim

- Left-click on Step + or Step – to optimize the selected.

- Repeat the process for X-, Y-, Z^2- shims

If the lock signal goes to the top of the window, left click on **Gain** (highlighted in Red) and adjust the lock level downward by left clicking on **Step -.**

△ Solvent	Description
Acetic	acetic acid–d4
Acetone	acetone–d6
C6D6	benzene–d6
CD2Cl2	methylenechloride–d2
CD3CN	acetonitrile–d3
CD3CN_SPE	LC–SPE Solvent (Acetonitrile)
CDCl3	chloroform–d
CH3CN+D2O	HPLC Solvent (Acetonitril/D2O)
CH3OH+D2O	HPLC Solvent (Methanol/D2O)
D2O	deuteriumoxide
DEE	diethylether–d10
Dioxane	dioxane–d8
DME	dimethylether–d6
DMF	dimethylformamide–d7
DMSO	dimethylsulfoxide–d6
EtOD	ethanol–d6
H2O+D2O	90%H2O and 10%D2O
MeOD	methanol–d4
Pyr	pyridine–d5
THF	tetrahydrofurane–d8
Tol	toluene–d8

Solvents table

OK   Cancel

(6) <u>Tuning the probe.</u>

The DPX-250 is equipped with a QNP probe thus, for the most part, eliminating the need to tune the probe.   This probe will automatically switch between, ^{1}H, ^{13}C, ^{31}P, and ^{15}N.

To automatically tune the probe on the DPX-400

       type: ***atma***

(7) **Checking Data Acquisition Parameters:**

After the instrument has been shimmed on your sample click on the Green Square in the upper right-hand corner of the screen to return to the <u>Acquisition Window</u>.

> Click on the **AcquPars** tab highlighted in blue.

From the panel of acquisition parameters, shown below:

- Set the desired pulse program (highlighted in red)

- Set the appropriate spectral window [**<u>sw</u>**] (highlighted in yellow)

- Choose the Number of datapoints [**<u>td</u>**] (highlighted in blue)
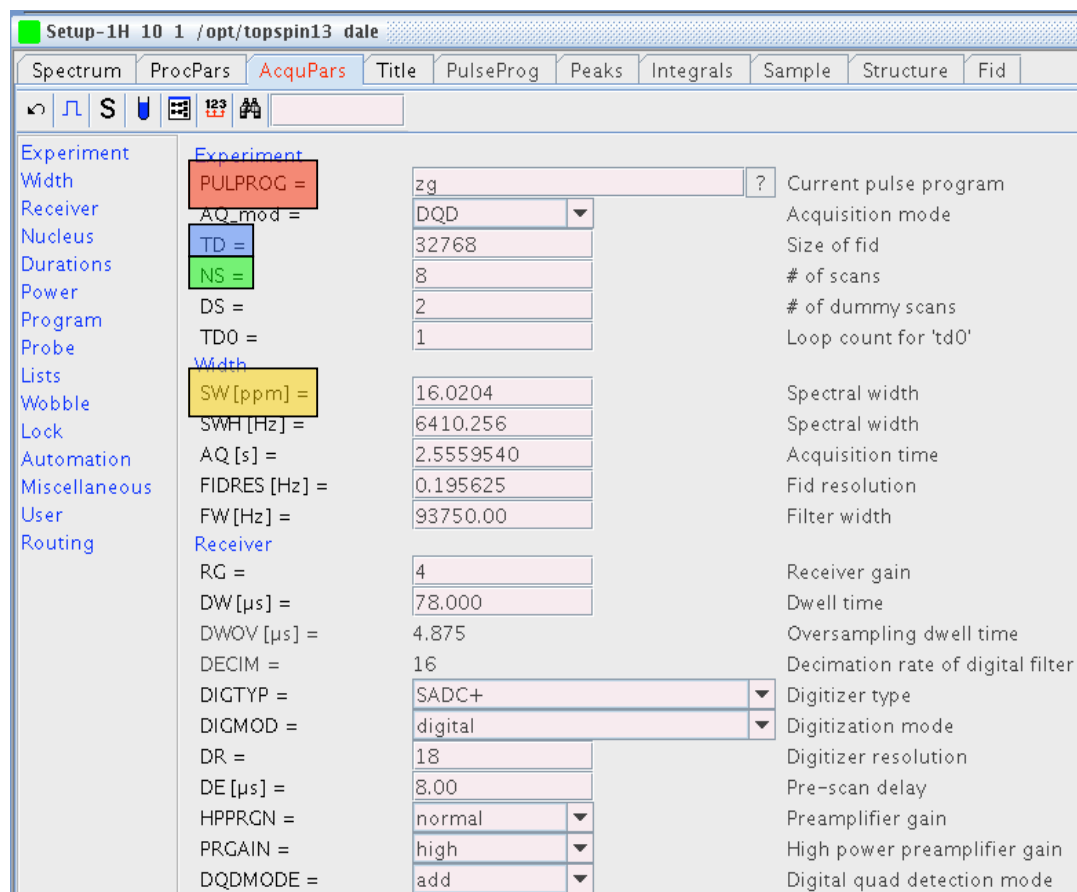
    > The number of data points, in concert with the spectral window, will determine your spectral resolution.

- Set the number of scans [**<u>ns</u>**] (highlighted in green)

    > Dilute samples require more scans!
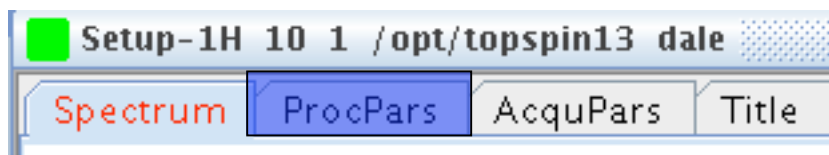
- Set O1 [advanced users only]
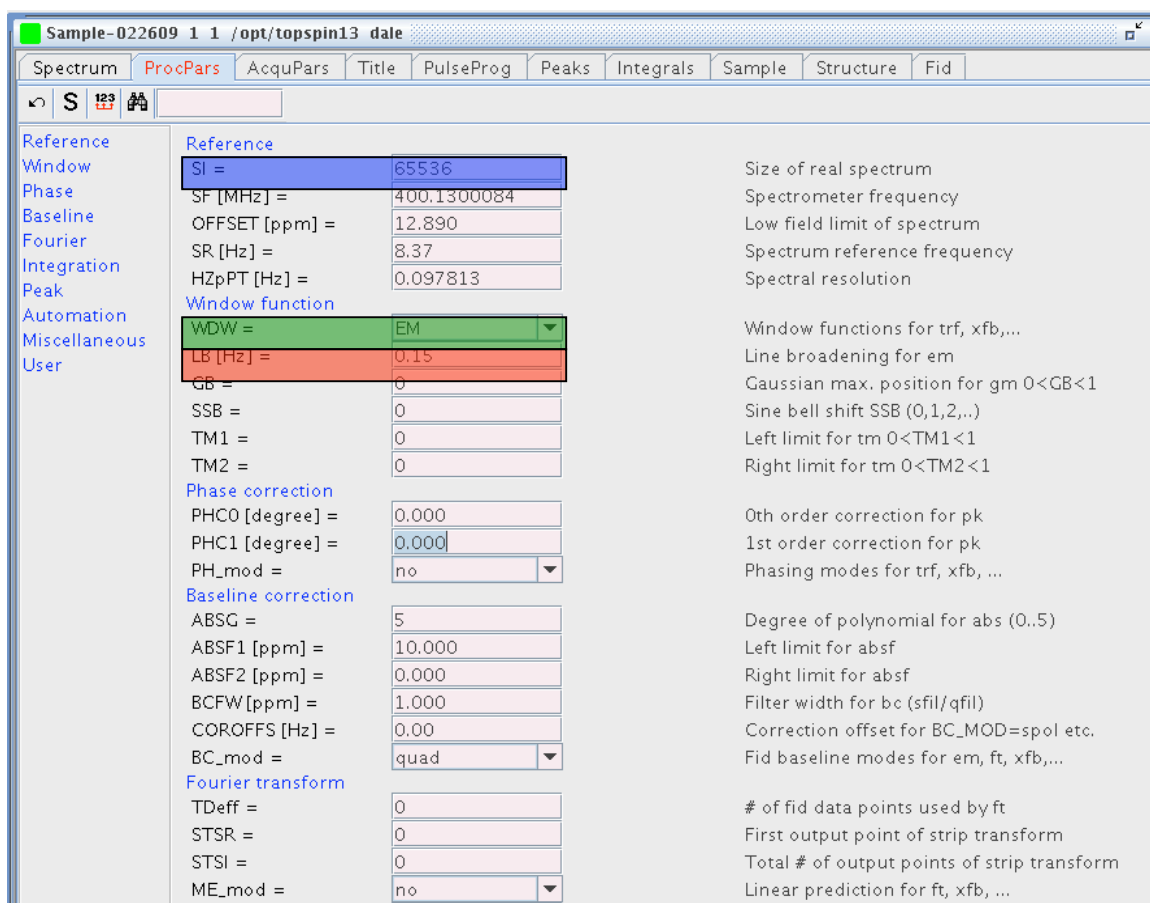
(8) Data ACQUISITION

type: *rga* (to automatically adjust the receiver gain)

type: *zg*

(9) Data Processing

Click on the **ProcPars** tab to open up a table showing the processing parameters.

Sample-022609 1 1 /opt/topspin13 dale

Spectrum | ProcPars | AcquPars | Title | PulseProg | Peaks | Integrals | Sample | Structure | Fid

Reference
Window
Phase
Baseline
Fourier
Integration
Peak
Automation
Miscellaneous
User

Reference

SI =	65536	Size of real spectrum
SF [MHz] =	400.1300084	Spectrometer frequency
OFFSET [ppm] =	12.890	Low field limit of spectrum
SR [Hz] =	8.37	Spectrum reference frequency
HZpPT [Hz] =	0.097813	Spectral resolution

Window function

WDW =	EM	Window functions for trf, xfb,...
LB [Hz] =	0.15	Line broadening for em
GB =	0	Gaussian max. position for gm 0<GB<1
SSB =	0	Sine bell shift SSB (0,1,2,..)
TM1 =	0	Left limit for tm 0<TM1<1
TM2 =	0	Right limit for tm 0<TM2<1

Phase correction

PHC0 [degree] =	0.000	0th order correction for pk
PHC1 [degree] =	0.000	1st order correction for pk
PH_mod =	no	Phasing modes for trf, xfb, ...

Baseline correction

ABSG =	5	Degree of polynomial for abs (0..5)
ABSF1 [ppm] =	10.000	Left limit for absf
ABSF2 [ppm] =	0.000	Right limit for absf
BCFW [ppm] =	1.000	Filter width for bc (sfil/qfil)
COROFFS [Hz] =	0.00	Correction offset for BC_MOD=spol etc.
BC_mod =	quad	Fid baseline modes for em, ft, xfb,...

Fourier transform

TDeff =	0	# of fid data points used by ft
STSR =	0	First output point of strip transform
STSI =	0	Total # of output points of strip transform
ME_mod =	no	Linear prediction for ft, xfb, ...

- Select dataset size <u>AFTER</u> Fourier transformation (**si**).

- Choose the apodization window function (WDW, <u>usually **em**</u>)

- Select linebroadening [**lb**] (approximately 0.2 Hz for ^{1}H).

- Type: [**efp**]

> This is actually a combination of three commands which will **E**xpenontially multiply your FID, **F**ourier transform the FID, and **P**hase correct the transformed spectrum.

Even though you just finished phasing your data, for a variety of reasons you may wish to touch up the phasing.

It is important to note that **zero-order (0)** phase correction will have a linear effect across the entire spectrum. By way of contrast, **first-order (1)** will have very little effect near the pivot point of the spectrum (shown by the red vertical line of the page following) and increasingly more effect at points further from the pivot point.
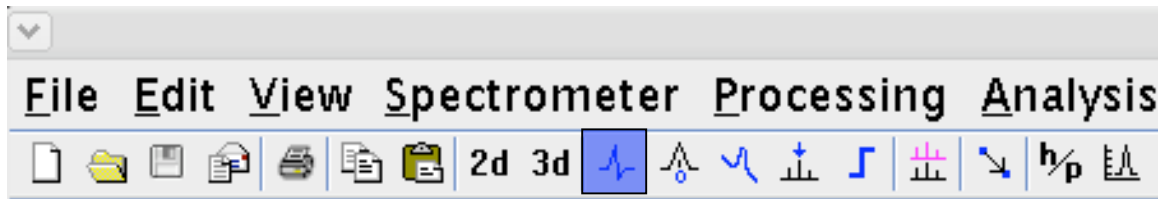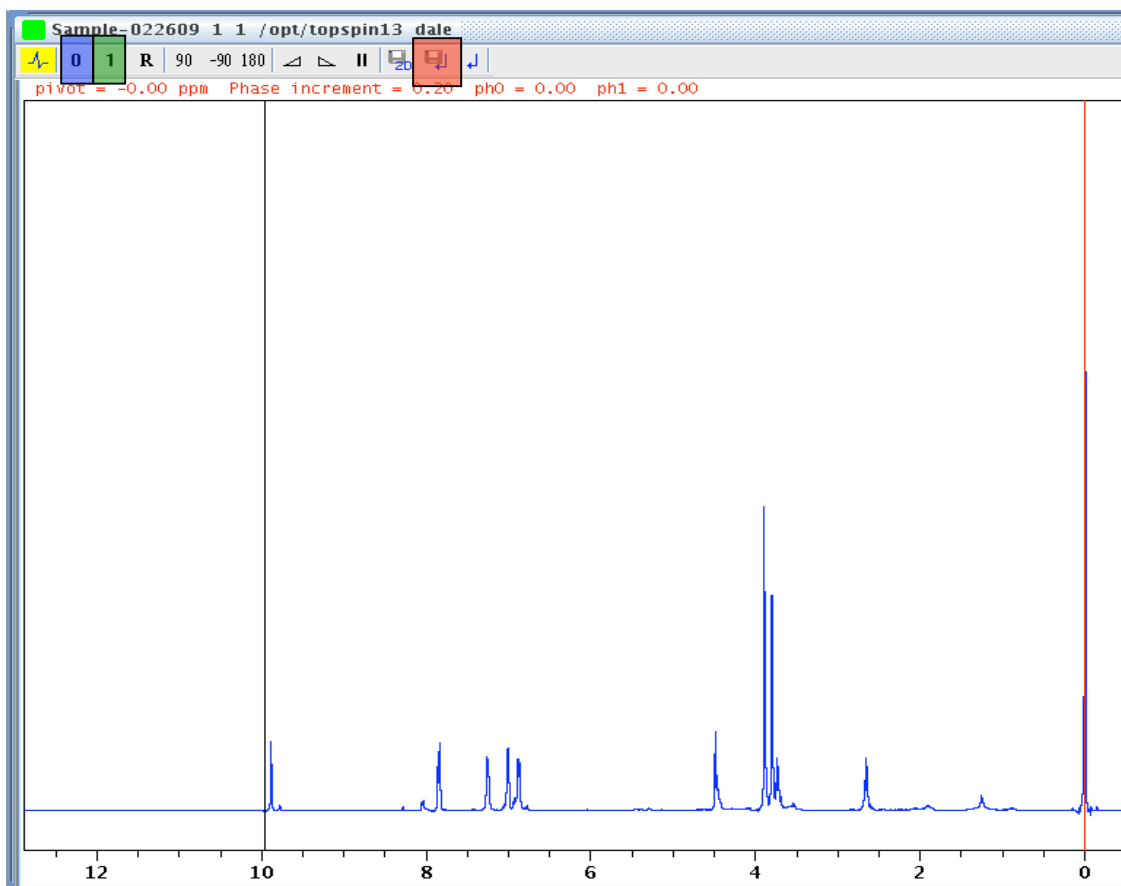
- Click on the phasing icon (highlighted in blue above.)

Zero-order phase correction:

Place the cursor over the zero-order phase tab, highlighted in blue on the panel blow. Press and hold down the left mouse button. Notice as you drag the mouse up or down the phase of the spectrum changes. Dragging the mouse, optimize the phase of the spectrum in the area immediately adjacent to the pivot point (the red, vertical line).

First-order phase correction:

Place the cursor on the first-order phase tab, highlighted in green, below. In a like manner to that used to adjust the zero-order phase, hold down the left mouse button while dragging the mouse up or down as needed to optimize the phase of the spectrum furthest away from the pivot point.

After you have phase corrected the spectrum you must save the phase angles that you have set during the above procedure.

- Click on the "floppy disk" icon, highlighted in red in order to save the phase angles.

(10) **Peak picking the peaks of interest**.

- Click on the Peak-Pick icon highlighted in red below.

- Place the cursor to the left of the most down-field peak of interest and above the highest peak of interest.

- Press and hold the left mouse button and drag the mouse diagonally across the spectral region of interest.

The green "box" indicates all of the peaks that will be labeled when the mouse button is released.
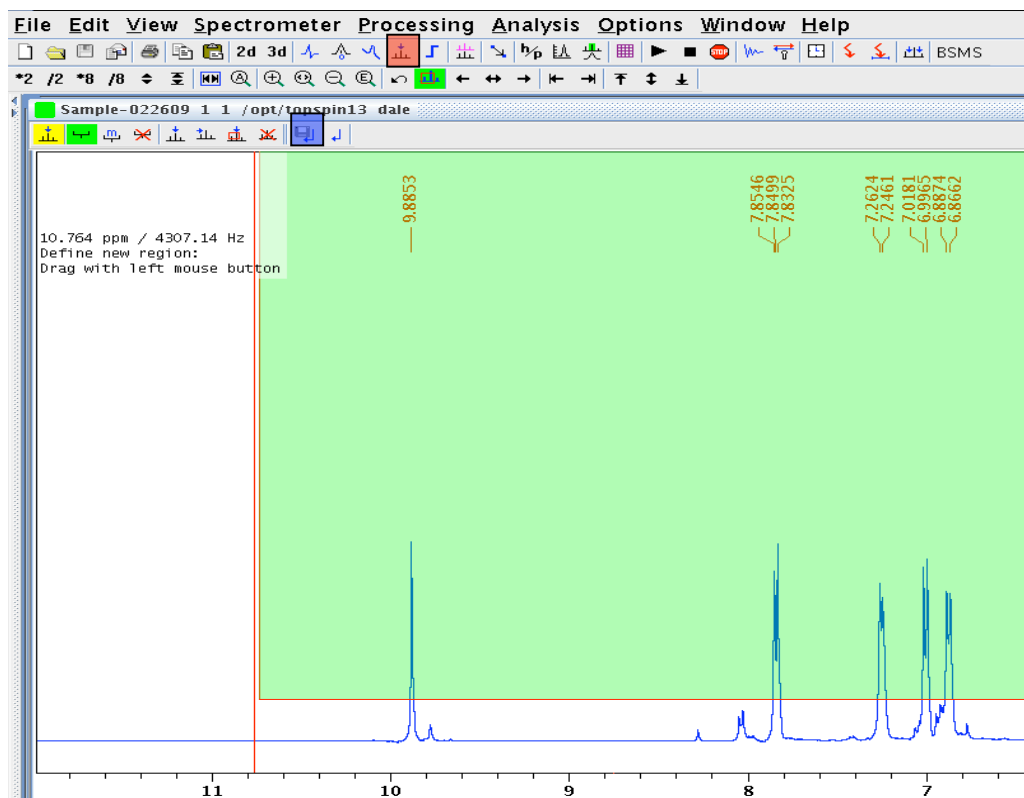
- Don't forget to click on the "floppy disk" icon, highlighted in blue, to save the peak labels to a peak list.
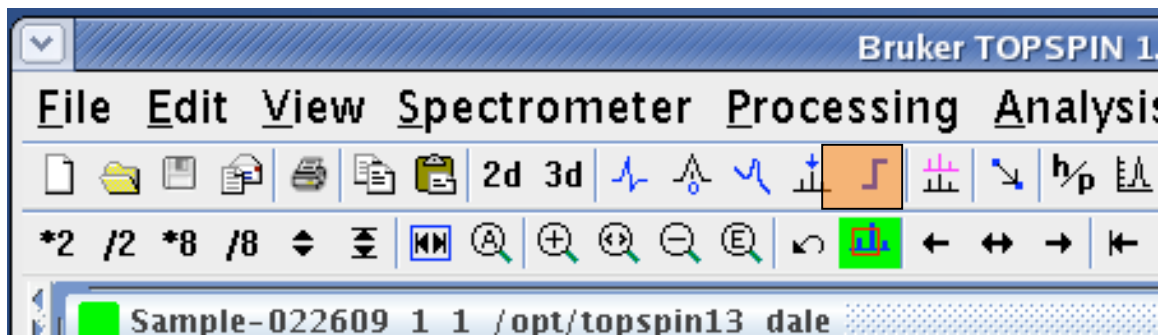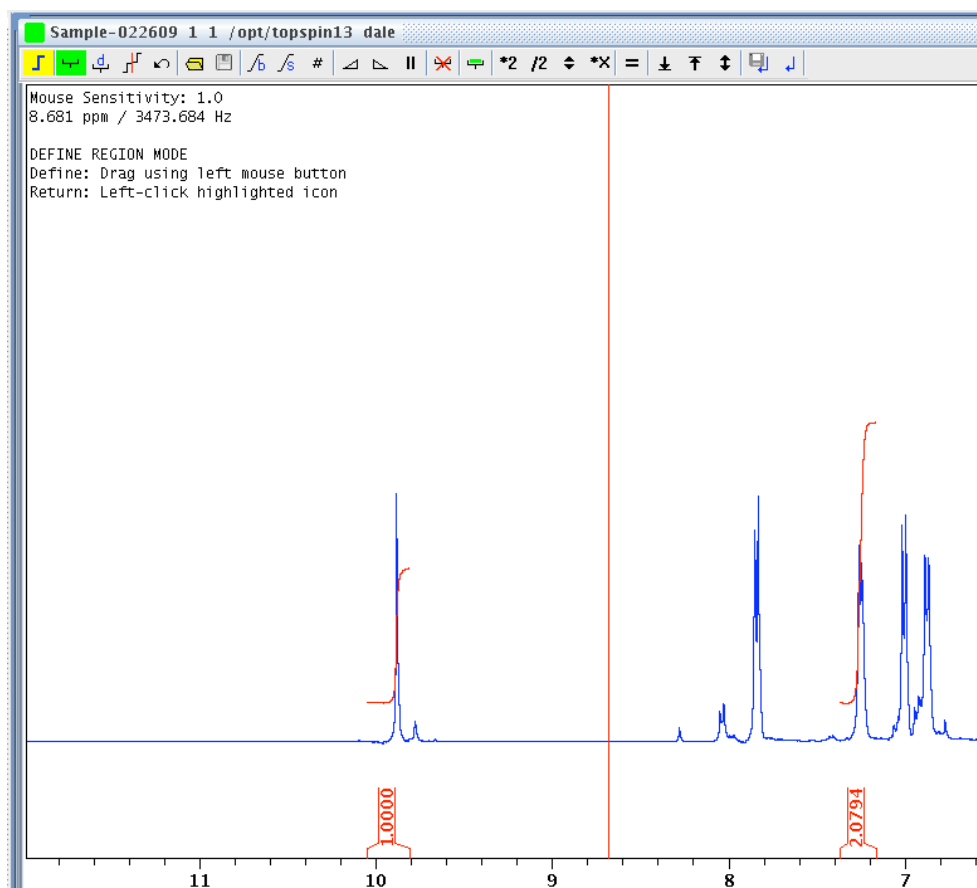
(11) Calibrating the spectrum

- Click on the (red) calibration icon show below.

- Left click on the peak of interest.

- Enter the chemical shift (PPM) in the dialog box.

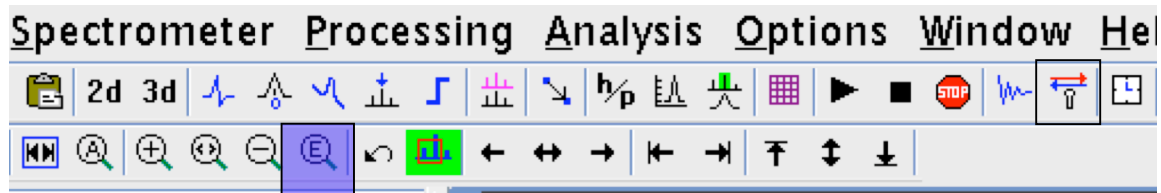- Click "OK".
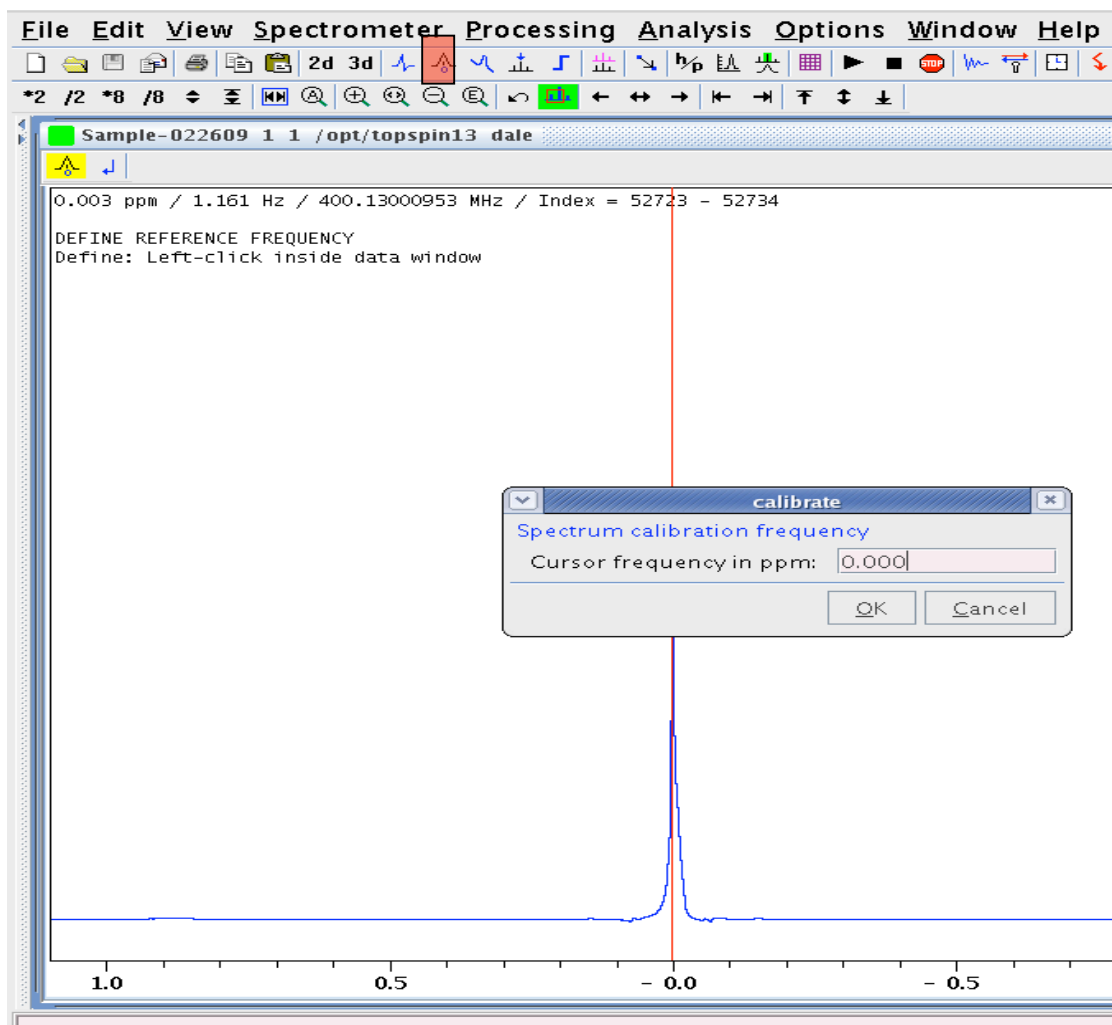
(12) Integrating the spectrum

- Click on the blue integral-shaped icon on the panel highlighted below.

- Place the cursor downfield of the first peak of interest.

- Press the left mouse button and drag the mouse up-field of the peak of interest.

- Left-click to return

- Repeat for each additional peak/region of interest.

- Place the cursor within the integral label of any peak you wish to calibrate and press the right mouse button.

- Select "calibrate".

- Enter the desired value of the selected integral.

- Click on the "floppy disk" icon to save the integrals.

(13) Plotting data:

- Click on the "Expanded Zoom" icon highlighted in blue.

- Enter the desired downfield and upfield plot limits.
  For example, 10.00 and -1.00 ppm

- Place the cursor anywhere in the window showing the spectrum and right click.

- Select **Display Properties** .

- Pick the features that you want to appear when you plot your data.

- Click on the "printer". By default the output will appear exactly as it appears on the screen.

- Select the desired printer.

- Click "OK"